

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2001-072490

(43)Date of publication of application : 21.03.2001

(51)Int.Cl.

C30B 23/00

C30B 29/36

(21)Application number : 11-243878

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(22)Date of filing : 30.08.1999

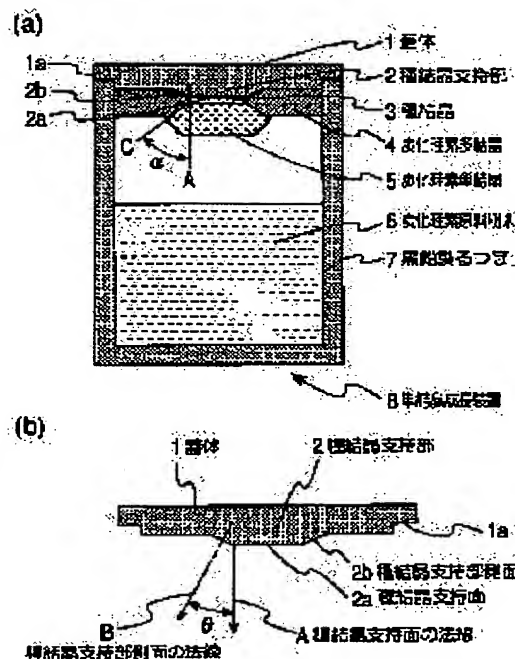
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(54) GROWING APPARATUS FOR SINGLE CRYSTAL AND PRODUCTION METHOD

(57)Abstract:

PROBLEM TO BE SOLVED: To readily enlarge the diameter of a growing single crystal by suppressing the obstruction of the growth by a polycrystal when growing the single crystal of silicon carbide or the like by a sublimation recrystallization method.

SOLUTION: The center part of the lower surface of a lid body 1 facing to the raw material powder 6 for silicon carbide, arranged in a crucible 7 is protruded to provide a seed crystal-supporting part 2, and the seed crystal 3 is attached on the supporting surface 2a. The seed crystal-supporting part 2 has a side surface 2b tilted to the supporting surface 2a side so as to have a contracted diameter, and the angle formed by the normal line of the side surface 2b and the normal line of the supporting surface 2a is regulated so as to be 20-80° to prevent polycrystal 4 growing on the side surface 2b from obstructing the growth of the single crystal 5 on a seed crystal 3, and further to prevent the side surface of the single crystal 5 from being thermally etched. The single crystal 5 can grow while expanding the diameter in the outer direction.



LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision
of rejection]

[Date of requesting appeal against examiner's
decision of rejection]

[Date of extinction of right]

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CLAIMS

[Claim(s)]

[Claim 1] Make the raw material and seed crystal of a single crystal to grow up counter in a container, and it arranges. It is equipment which carries out heating sublimation of the above-mentioned raw material, and grows up a single crystal on the above-mentioned seed crystal. While making the seed crystal supporter which a part of above-mentioned container internal surface which counters the above-mentioned raw material is made to project to the above-mentioned raw material side, and supports the above-mentioned seed crystal, and nothing and this seed crystal supporter into the abbreviation trapezoid configuration where a side face inclines Growth equipment of the single crystal characterized by making it the include angle theta of the normal in the center section of this inclining side face and the normal of the above-mentioned back face to make turn into 80 or less degrees 20 degrees or more.

[Claim 2] Growth equipment of the single crystal according to claim 1 which made the above-mentioned side face of the above-mentioned seed crystal supporter the shape of the shape of a convex, and a concave surface.

[Claim 3] Claims 1 or 2 which made the minor diameter the path of the above-mentioned back face of the above-mentioned seed crystal supporter from the path of the above-mentioned seed crystal, abbreviation identitas, or the above-mentioned seed crystal are growth equipment of the single crystal of a publication either.

[Claim 4] 3 is [claim 1 whose above-mentioned single crystal is silicon carbide monocrystal thru/or] growth equipment of the single crystal of a publication either.

[Claim 5] In the approach of making the raw material and seed crystal of a single crystal to grow up countering in a container, arranging, carrying out heating sublimation of the above-mentioned raw material, and growing up a single crystal on the above-mentioned seed crystal While making the seed crystal supporter which a part of above-mentioned container internal surface which counters the above-mentioned raw material is made to project to the above-mentioned raw material side, and supports the above-mentioned seed crystal, and nothing and this seed crystal supporter into the abbreviation trapezoid configuration where a side face inclines The manufacture approach of the single crystal characterized by making it the include angle theta of the normal in the center section of this inclining side face and the normal of the above-mentioned back face to make turn into 80 or less degrees 20 degrees or more.

[Claim 6] The manufacture approach of the single crystal according to claim 5 which made the above-mentioned side face of the above-mentioned seed crystal supporter the shape of the shape of a convex, and a concave surface.

[Claim 7] The manufacture approach of the single crystal according to claim 5 or 6 which made the minor diameter the path of the above-mentioned back face of the above-mentioned seed crystal supporter from the path of the above-mentioned seed crystal, abbreviation identitas, or the above-mentioned seed crystal.

[Claim 8] 7 is [claim 5 whose above-mentioned single crystal is silicon carbide monocrystal thru/or] the manufacture approach of the single crystal a publication either.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the growth equipment and the manufacture approach of a single crystal which are used in order to grow up single crystals, such as silicon carbide.

[0002]

[Description of the Prior Art] Silicon carbide monocrystal is excellent in thermal and chemical property, and attracts attention as substrate ingredients for semiconductor device production, such as a power device. Silicon carbide monocrystal is obtained by the sublimation recrystallizing method, makes the raw material and seed crystal of a single crystal to grow up counter generally, is arranged, carries out heating sublimation of the raw material, and grows up a single crystal on seed crystal. Drawing 5 shows an example of the growth equipment by the sublimation recrystallizing method, and single crystal growth equipment 18 has the lid 11 by which ** arrival is carried out to the crucible 17 made from a graphite with which it fills up with the silicon carbide raw material powder 16, and this crucible 17. If junction immobilization of the seed crystal 13 which consists of silicon carbide monocrystal is carried out and a lid 11 is made to carry out heating sublimation of the raw material powder 16, it will recrystallize in it on the seed crystal 13 with which the sublimation gas counters, and silicon carbide monocrystal 15 will grow up to be it.

[0003] By the way, as a silicon-carbide-monocrystal substrate for semiconductor device production, although current and a thing with a diameter of about 2 inches are marketed, in order to raise mass-production nature, the silicon-carbide-monocrystal substrate of the diameter of macrostomia is needed more. However, with the conventional growth equipment 18 shown in above-mentioned drawing 5, since seed crystal 13 is directly joined to a lid 11, the polycrystal 14 deposited on lid 11 front face of the seed crystal 13 circumference and the single crystal 15 which grows on seed crystal 13 contact. For this reason, there was a problem from which aperture expansion of a growth crystal is prevented.

[0004] Then, the growth equipment which the equipment for solving this problem is proposed variously, for example, covered the periphery of seed crystal with the diaphragm to JP,6-37353,B is indicated. He controls generating of polycrystal and is trying for a single crystal to grow only on seed crystal by holding a diaphragm from seed crystal to an elevated temperature with this growth equipment. Moreover, there is growth equipment which formed the height in the lid and was used as the seed crystal supporter so that it may be indicated by JP,1-305898,A, JP,10-36195,A, etc. Like drawing 6, by using as the seed crystal supporter 12 the height formed in the center of an inferior surface of tongue of a lid 11, seed crystal 13 is arranged on it, and the polycrystal 14 deposited on the perimeter of the seed crystal supporter 12 delays the timing in contact with the single crystal 15 which grows on seed crystal 13, and is raising the aperture dilation ratio of a growth crystal with this equipment.

[0005]

[Problem(s) to be Solved by the Invention] However, with the equipment of JP,6-37353,B, the aperture of the single crystal obtained is restricted by the diameter of opening of a diaphragm,

and cannot enlarge an aperture dilation ratio enough. Moreover, equipment structure is complicated and there is a problem which manufacture of equipment takes cost and time amount. On the other hand, with the equipment of JP,1-305898,A and JP,10-36195,A, in order that polycrystal 14 might contact a single crystal 15 if growth progresses as shown in drawing 6 although it is possible to delay the timing to which polycrystal contacts a single crystal, there was fault that expansion of the aperture beyond it was obstructed.

[0006] This invention is made in view of the above-mentioned actual condition, and in growing up single crystals, such as silicon carbide, by the sublimation recrystallizing method, the purpose controls the growth inhibition by polycrystal, and is simple to offer the growth equipment and the manufacture approach of a single crystal that aperture expansion of the single crystal which grows can be aimed at.

[0007]

[Means for Solving the Problem] The growth equipment of the single crystal of claim 1 of this invention is equipment which makes the raw material and the seed crystal of a single crystal to grow up counter in a container, and it arranges [equipment], carries out heating sublimation of the above-mentioned raw material, and grows up a single crystal on the above-mentioned seed crystal, makes a part of the above-mentioned container internal surface which counters the above-mentioned raw material project to an above-mentioned raw material side, and has made with the seed-crystal supporter which supports the above-mentioned seed crystal. This seed crystal supporter is the abbreviation trapezoid configuration where a side face inclines, and it is formed so that it may become 80 or less degrees 20 degrees or more about the include angle theta of the normal in the center section of this inclining side face, and the normal of the above-mentioned back face to make.

[0008] Since it considered as the inclined plane which turns the above-mentioned side face to a back-face side, and reduces the diameter of it in this invention although polycrystal grew on the side face of the above-mentioned seed crystal supporter when the single crystal was grown up using the above-mentioned equipment, the growth direction of the polycrystal which grows up to be this inclining side face turns into the direction of that normal. That is, polycrystal will grow so that it may escape outside to the growth direction (the direction of the normal of the above-mentioned back face) of the single crystal which grows on the above-mentioned seed crystal, and for this reason, the growth inhibition by polycrystal like a configuration (refer to drawing 5) before soon joined to the lid can be controlled. Since it can grow up while this effectiveness is high and a single crystal is expanded outside if an include angle theta is especially made into 20 degrees or more, an aperture dilation ratio becomes large.

[0009] On the other hand, conventionally which formed the convex seed crystal supporter, with a configuration (refer to drawing 6), there is no growth inhibition by polycrystal until the polycrystal which grows on a lid contacts a single crystal, but since the side face of a single crystal is put to an elevated temperature in the meantime, it is eaten away by heat etching and an aperture dilation ratio falls. On the other hand, in this invention, polycrystal accumulates from immediately after initiation of single crystal growth on the inclining above-mentioned side face, the single crystal with which this grows is contacted, and a side face is protected. In order to acquire this effectiveness, it can be good to make an include angle theta into 80 or less degrees, it can prevent heat etching, and can make aperture expand.

[0010] Therefore, if the above-mentioned include angle theta is made into 80 or less degrees 20 degrees or more, the above-mentioned aperture expansion effectiveness is remarkable, and heat etching will be prevented in contact with the single crystal side face in which it grows up, without polycrystal blocking growth of a single crystal. Consequently, the aperture dilation ratio of a single crystal can be raised sharply, moreover, an equipment configuration is easy and manufacture is also easy an equipment configuration.

[0011] With the equipment of claim 2, the above-mentioned side face of the above-mentioned seed crystal supporter is formed the shape of a convex, and in the shape of a concave surface. Although the above-mentioned seed crystal supporter is generally formed so that the cross section of the above-mentioned side face may become straight line-like, an abbreviation center section can also make the above-mentioned side face configurations, such as the shape of a

convex which bulges in the method of the outside of the direction of a path, and the shape of a concave surface which carries out a cavity to the method of the inside of the direction of a path. In this case, although the above-mentioned include angle θ changes with parts, the same effectiveness will be acquired if the include angle θ in a side-face center section except the above-mentioned back-face side edge section or the above-mentioned container internal-surface side edge section is in the above-mentioned range at least.

[0012] Let the path of the above-mentioned back face of the above-mentioned seed crystal supporter be a minor diameter with the equipment of claim 3 from the path of the above-mentioned seed crystal, abbreviation identitas, or the above-mentioned seed crystal. If the path of the above-mentioned back face is larger than the above-mentioned seed crystal, since polycrystal will accumulate on the above-mentioned back face around the above-mentioned seed crystal, in order to acquire the effectiveness of the inclining above-mentioned side face, it is desirable to make the above-mentioned seed crystal and the above-mentioned back face into the almost same magnitude, or to form the above-mentioned back face small a little.

[0013] Let the above-mentioned single crystal be silicon carbide monocrystal with the equipment of claim 4. It is useful as a substrate for semiconductor device production, and moreover, conventionally, since diameter[of macrostomia]-izing was difficult for silicon carbide monocrystal, the effectiveness at the time of applying this invention is remarkable [silicon carbide monocrystal].

[0014] Invention of claim 5 makes a part of above-mentioned container internal surface which counters the above-mentioned raw material project to the above-mentioned raw material side about the manufacture approach of a single crystal in the approach of making the raw material and the seed crystal of a single crystal grown up into a container countering, arranging, carrying out heating sublimation of the above-mentioned raw material, and growing up a single crystal on the above-mentioned seed crystal, and carries out support immobilization in the above-mentioned seed crystal a seed-crystal supporter, nothing, and on it. This seed crystal supporter is made into the abbreviation trapezoid configuration where a side face inclines, and it is made for the include angle θ of the normal in the center section of this inclining side face and the normal of the above-mentioned back face to make to turn into 80 or less degrees 20 degrees or more at this time.

[0015] According to the above-mentioned approach, the same effectiveness as above-mentioned claim 1 is acquired, and the aperture dilation ratio of a single crystal can be sharply raised by the simple approach.

[0016] Like the approach of claim 6, the above-mentioned side face of the above-mentioned seed crystal supporter can also be formed the shape of a convex, and in the shape of a concave surface. Even if such, the same aperture expansion effectiveness is acquired by each by making it the above-mentioned include angle θ in a center section serve as the above-mentioned range.

[0017] Let the path of the above-mentioned back face of the above-mentioned seed crystal supporter be a minor diameter by the approach of claim 7 from the path of the above-mentioned seed crystal, abbreviation identitas, or the above-mentioned seed crystal. Thereby, an operation of the above-mentioned side face which prevents and inclines that polycrystal accumulates on the above-mentioned back face can be demonstrated effectively.

[0018] Let the above-mentioned single crystal be silicon carbide monocrystal by the approach of claim 8. Silicon carbide monocrystal is useful as a substrate for semiconductor device production, and its utility value by diameter[of macrostomia]-izing is large.

[0019]

[Embodiment of the Invention] Hereafter, the gestalt of 1 operation of this invention is explained to a detail based on a drawing. Drawing 1 (a) is the outline block diagram of the growth equipment of the single crystal which applied this invention, and explains the case where the single crystal to grow up is made into silicon carbide monocrystal here. The growth equipment 8 of a single crystal has the crucible 7 made from a graphite and lid 1 as a container among drawing. A crucible 7 is the bottle object of upper limit opening, and the bottom half circles are filled up with the silicon carbide raw material powder 6 used as a raw material. As shown in

drawing 1 (b), the lid 1 is making the periphery edge with flange 1a of thin meat a little, and when it attaches in a crucible 7, it has made it as [seal / in contact with the upper limit edge of a crucible 7 / flange 1a / this] (drawing 1 (a)).

[0020] Next, the configuration of the lid 1 which is the description part of this invention is explained. In drawing 1 (a) and (b), a lid 1 makes the center of an inferior surface of tongue (container internal surface) project below (raw material powder 6 side), and is forming seed crystal 3 with the seed crystal supporter 2 for carrying out support immobilization. This crystal supporter 2 is made with the configuration whose diameter is gradually reduced towards the back-face 2a side which touches seed crystal 3 from a lid 1 inferior-surface-of-tongue side so that it is an abbreviation trapezoid configuration, and side-face 2b may have the raw material powder 6 and an inclination and may counter. Back-face 2a to which seed crystal 3 is fixed is formed in lid 1 inferior surface of tongue and parallel.

[0021] Are specifically good to make side-face 2b incline so that the include angle theta of the normal A of back-face 2a and the normal B of side-face 2b (henceforth whenever [tilt-angle / theta]) to make may turn into (drawing 1 (b)) and 80 degrees or less 20 degrees or more. By this It can control that growth of silicon carbide monocrystal 5 is checked with the silicon carbide polycrystal 4 which grows up to be the perimeter of seed crystal 3, and the side face of a single crystal 5 can be protected from heat etching with polycrystal 4, and the aperture dilation ratio of the growth crystal 5 can be enlarged effectively. If theta is [whenever / tilt-angle] smaller than 20 degrees, it will become conventionally close to a configuration and the growth inhibition by surrounding polycrystal shown in above-mentioned drawing 6 will become large. moreover, when theta was [whenever / tilt-angle] larger than 80 degrees, it was shown in above-mentioned drawing 7 -- it becomes conventionally close to a configuration, heat etching of the single crystal periphery section is carried out, and the aperture expansion effectiveness decreases. theta is decided by magnitude of both growth rate of polycrystal 4, and growth rate of the direction of single crystal 5 side face who grow on the outskirts whenever [tilt-angle / from which the aperture expansion effectiveness serves as max]. Although both magnitude changed with the growth conditions at that time, and the structures of growth equipment, when this invention persons were the range of the growth conditions which are growth equipment of the structure shown in drawing 1 (a), and are shown below, theta found out that the aperture expansion effectiveness was acquired in 20 to 80 degrees whenever [tilt-angle]. More preferably, it is good to make theta into the range of 25 to 65 degrees whenever [tilt-angle], and it can heighten the above-mentioned effectiveness further. In addition, with the gestalt of this operation, side-face 2b of the crystal supporter 2 is formed so that a cross section may become straight line-like, and theta is [whenever / tilt-angle] fixed.

[0022] Here, as seed crystal 3 for growing up silicon carbide monocrystal 6, the silicon carbide monocrystal manufactured with the Acheson process or the silicon carbide monocrystal grown up by the sublimating method from the Atchison crystal is usually used. Seed crystal 3 comes to process these silicon carbide monocrystal in the shape of [predetermined] a wafer type, and junction immobilization is carried out on back-face 2a of the seed crystal supporter 2. this time -- the path of back-face 2a -- the path of seed crystal 3, and abbreviation -- or [being same] -- or it can be good to make it become small a little, and it can demonstrate the above-mentioned aperture expansion effectiveness more effectively. When the path of back-face 2a is too larger than the path of seed crystal 3, polycrystal accumulates on back-face 2a of the seed crystal 3 circumference, and there is a possibility of having a bad influence on aperture expansion.

[0023] In addition, although not restricted, if especially the protrusion height (height of the vertical direction of drawing) of the seed crystal supporter 2 is too small not much, the polycrystal 4 which grows on a lid 1 will catch up with growth of a single crystal 5, and it will become close to the condition (whenever [tilt-angle] condition of $\theta = 0$) that there is no seed crystal supporter 2. Moreover, since there is a problem of the temperature distribution of the circumference of seed crystal 3 changing when protrusion height is too large, it is good to set up suitably according to an equipment configuration, the magnitude of seed crystal 3, etc.

[0024] In growing up a single crystal using the above-mentioned equipment, like drawing 1 (a), it

joins to back-face 2a of the crystal supporter 2 with adhesives, and heats seed crystal 3 with heating apparatus (****), such as an induction coil arranged on the perimeter of a crucible 7. At this time, beyond the sublimation temperature (usually about about 2000-2500 degrees C) of silicon carbide, the raw material powder 6 establishes a temperature gradient in a crucible 7 so that it may become temperature with seed crystal 3 lower than the raw material powder 6. The ambient atmosphere in a crucible 7 is made into inert gas ambient atmospheres, such as argon gas, and a pressure is good to consider as 0.1 - 10Torr extent. By this, the sublimation gas of the raw material powder 6 occurs, and it is spread upwards, and recrystallizes on the low-temperature seed crystal 3 more.

[0025] Thus, it is possible to make the aperture expansion include angle alpha of the silicon carbide monocrystal 6 obtained (include angle of the boundary line C of a single crystal 5 and polycrystal 4 and the normal A of back-face 2a to make) larger than before by making side-face 2b of the crystal supporter 2 into an inclined plane, and making theta (include angle of the normal A of back-face 2a and the normal B of side-face 2b to make) into 80 or less degrees 20 degrees or more whenever [tilt-angle]. In order that this may grow so that the polycrystal 4 which grows in the direction of the normal B of inclining side-face 2b may escape outside to the growth direction (the direction of the normal of the above-mentioned back face) of the single crystal 5 which grows on seed crystal 3 [0026] which can moreover aim at aperture expansion of the single crystal 5 which grows simply by both effectiveness of these by preventing heat etching since the side face of a single crystal 5 is protected by this polycrystal 4, that the growth inhibition by polycrystal 4 is prevented, and The gestalt of operation of the 2nd of this invention is shown in drawing 2 . In the gestalt of implementation of the above 1st, side-face 2b of the crystal supporter 2 is formed so that a cross section may become straight line-like, and although theta is [whenever / tilt-angle] fixed, an abbreviation center section can also make side-face 2b the shape of a convex which bulges in the method of the outside of the direction of a path like drawing 2 . In this case, it is making it theta become 80 or less degrees from the back-face 2a side 20 degrees or more whenever [tilt-angle] in the center section except the back-face 2a side edge section and the lid 1 side-edge section, although theta's becomes large gradually whenever [tilt-angle] towards a lid 1 side, and the same effectiveness is acquired. Moreover, as the gestalt of operation of the 3rd of this invention is shown in drawing 3 , an abbreviation center section can also form side-face 2b in the shape of [which carries out a cavity to the method of the inside of the direction of a path] a concave surface. Although it is the same and theta becomes small gradually whenever [tilt-angle] towards the lid 1 side from the back-face 2a side also in this case, in the center section except the back-face 2a side edge section and the lid 1 side-edge section, the same effectiveness is acquired by making it theta become 80 or less degrees 20 degrees or more whenever [tilt-angle].

[0027] Moreover, although the configuration of the crystal supporter 2 and seed crystal 3 has a common round shape, it cannot be restricted circularly but can also be made into polygons, such as other configurations, for example, a square, and a hexagon. There is not necessarily no need of making in agreement the configurations of the crystal supporter 2 and seed crystal 3, for example, the polygonal seed crystal 3 can be made to be able to support on the circular crystal supporter 2, or it can also make the circular seed crystal 3 support on the polygonal crystal supporter 2 conversely at this time.

[0028]

[Example] In order to check the effectiveness of this invention, the single crystal growth equipment 8 of above-mentioned drawing 1 was used, and silicon carbide monocrystal 5 was grown up. Adhesives were used and stuck on back-face 2a of the seed crystal supporter 2 formed in the lid 1 after fabricating this with a diameter of 10mm in the shape of a wafer, using the Atchison crystal as seed crystal 3. Whenever [10mm and tilt-angle], the outer diameter of back-face 2a formed [theta (include angle of the normal A of back-face 2a, and the normal B of side-face 2b to make)] the seed crystal supporter 2 so that it might become 30 degrees. Attachment immobilization was carried out and the lid 1 which was filled up with commercial silicon carbide abrasives (mean particle diameter of 80 micrometers) as silicon carbide raw material powder 6, and stuck seed crystal 3 in the crucible 7 at the upper limit opening was

heated all over the furnace for growth of ****. Under the present circumstances, first, the inside of a crucible 7 was permuted by the argon gas ambient atmosphere by the evacuation system of ****, after raising whenever [crucible 7 internal-temperature] to growth temperature by ambient pressure force 700Torr, the ambient pressure force was decompressed to the growth pressure (10Torr), and growth was started. Heating was made into the induction-heating method by the induction coil of ****, the raw material powder 6 was heated at about 2200-2300 degrees C, and the single crystal 5 was grown up on the seed crystal 3 held to temperature lower than this. Growth time amount was made into 24 hours, and kept temperature and a pressure constant during growth. At the time of growth termination, first, after raising the ambient pressure force to 700Torr(s), temperature was reduced to ordinary temperature.

[0029] Then, the silicon carbide monocrystal 5 which removed drawing and a lid 1 and grew the furnace for growth to the crucible 7 on seed crystal 3 was evaluated. Evaluation was performed using the photograph of the vertical cutting plane which cut and photoed the obtained silicon carbide monocrystal 5 to the lengthwise direction (the vertical direction of drawing 1), measured the include angle which the boundary line C of silicon carbide monocrystal 5 and the silicon carbide polycrystal 4 and the normal A of back-face 2a make, and was made into the aperture expansion include angle alpha. Furthermore, the aperture expansion include angle alpha was measured, respectively about the silicon carbide monocrystal 5 which changed theta in 15 to 90 degrees whenever [tilt-angle / of the seed crystal supporter 2], and was grown up by the same approach. Based on these measurement result, the relation between theta and the aperture expansion include angle alpha was investigated whenever [tilt-angle], and it was shown in drawing 4 . In addition, at this time, two or more single crystals 5 by theta were grown up whenever [same tilt-angle], the sample of the vertical cutting plane of each single crystal 5 was created, and the aperture expansion include angle alpha of those right and left was measured, respectively. The aperture expansion include angle alpha was shown in drawing 4 with the width of face of dispersion by the part of this sample, and dispersion between samples.

[0030] The aperture expansion include angle alpha is increasing and the aperture expansion effectiveness of 40 degrees or more is acquired [whenever / tilt-angle / the aperture expansion include angle alpha] for the aperture expansion include angle alpha at theta= 25 degrees whenever [tilt-angle] 30 degrees or more by theta= 20 degrees as theta becomes large from 15 degrees whenever [tilt-angle] so that clearly [drawing 4]. That is, it turns out that it can grow up while growth of a single crystal 5 is not checked but a single crystal 5 is expanded more outside by enlarging theta whenever [tilt-angle], since the growth direction of the polycrystal 4 which grows on side-face 2b of the seed crystal supporter 2 inclines outside more. On the other hand, whenever [near the conventional equipment configuration of drawing 5 / tilt-angle], at theta= 15 degrees, sufficient effectiveness is not acquired but it is thought that the growth inhibition by the polycrystal 4 which approaches a single crystal 5 and grows has arisen.

[0031] On the other hand, if theta exceeds 40 degrees whenever [tilt-angle], at theta= 90 degrees, the aperture expansion include angle alpha will turn into about 25 degrees whenever [tilt-angle / from which the aperture expansion include angle alpha becomes small gradually, and serves as the conventional equipment configuration of drawing 6]. It turns out that this is considered for the side face of a single crystal 5 being exposed, and receiving heat etching in order that a single crystal 5 and polycrystal 4 may not touch, if theta becomes large too much whenever [tilt-angle], and the effectiveness of being touching at this extent to which the polycrystal 4 on side-face 2b does not bar growth of a single crystal 5, and preventing heat etching of a single crystal 5 is acquired. As mentioned above, in order for the polycrystal 4 on side-face 2b not to bar growth of a single crystal 5 and to protect the side face of a single crystal 5 from heat etching, it turns out [whenever / tilt-angle] that aperture is sharply expandable compared with the former that theta should just be in the range of 20 to 80 degrees.

[0032] In addition, although the gestalt of the above-mentioned implementation explained the case where silicon carbide monocrystal was grown up as a single crystal, it is easy to be natural even if it applies to growth of other single crystals which can grow by the sublimation recrystallizing method besides silicon carbide monocrystal.

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] The gestalt of operation of the 1st of this invention is shown, (a) is the outline sectional view of single crystal growth equipment, and (a) is the expanded sectional view of a lid.

[Drawing 2] It is the expanded sectional view of the lid in the gestalt of operation of the 2nd of this invention.

[Drawing 3] It is the expanded sectional view of the lid in the gestalt of operation of the 3rd of this invention.

[Drawing 4] It is drawing showing the relation between an include angle theta and the aperture expansion include angle alpha.

[Drawing 5] It is the outline sectional view of conventional single crystal growth equipment.

[Drawing 6] It is the outline sectional view of conventional single crystal growth equipment.

[Description of Notations]

1 Lid

2 Seed Crystal Supporter

2a Back face

2b Side face

3 Seed Crystal

4 Silicon Carbide Polycrystal

5 Silicon Carbide Monocrystal

6 Raw Material Powder

7 Crucible

8 Single Crystal Growth Equipment

[Translation done.]

(19)日本国特許庁 (J P)

(12) 公 開 特 許 公 報 (A)

(11)特許出願公開番号
特開2001-72490
(P2001-72490A)

(43)公開日 平成13年3月21日(2001.3.21)

(51)Int.Cl. ⁷	識別記号	F I	テマコード [*] (参考)
C 3 0 B 23/00		C 3 0 B 23/00	4 G 0 7 7
29/36		29/36	A

審査請求 未請求 請求項の数 8 O L (全 7 頁)

(21)出願番号 特願平11-243878

(22)出願日 平成11年8月30日(1999.8.30)

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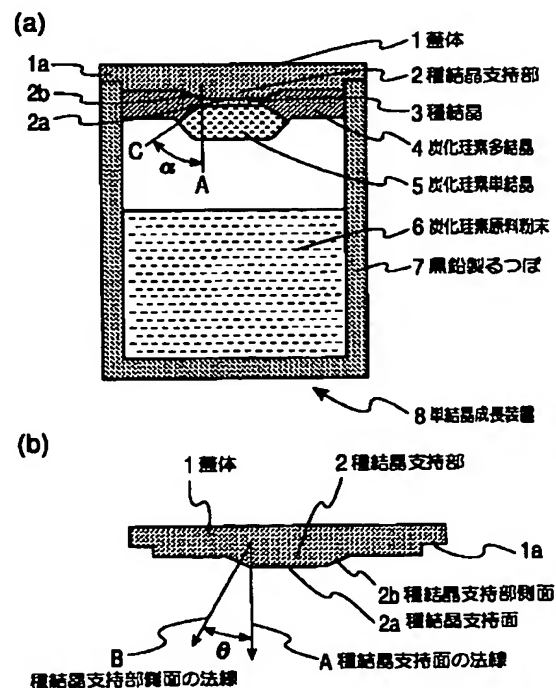
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(54)【発明の名称】 単結晶の成長装置および製造方法

(57)【要約】

【課題】 昇華再結晶法により炭化珪素等の単結晶を成長させるにあたり、多結晶による成長阻害を抑制して、簡便に、成長する単結晶の口径拡大を図る。

【解決手段】 るつぼ7内に配した炭化珪素原料粉末6に対向する蓋体1の下面中央部を突出させて種結晶支持部2とし、その支持面21上に種結晶3を接合する。種結晶支持部2は、支持面2a側へ向けて縮径するように側面22を傾斜させてあり、その傾斜する側面2bの法線と支持面2aの法線のなす角度を20度以上80度以下の範囲とすると、側面2b上に成長する多結晶4が、種結晶3上の単結晶5の成長を阻害することなく、しかも単結晶5の側面が熱エッチングされるのを防止する。よって、単結晶5が外側に拡大しつつ成長することができる。



【特許請求の範囲】

【請求項1】 容器内に、成長させる単結晶の原料と種結晶とを対向させて配置し、上記原料を加熱昇華させて上記種結晶上に単結晶を成長させる装置であって、上記原料に対向する上記容器内壁面の一部を上記原料側に突出させて、上記種結晶を支持する種結晶支持部となし、該種結晶支持部を、側面が傾斜する略台形形状とするとともに、この傾斜する側面の中央部における法線と上記支持面の法線とのなす角度 θ が20度以上80度以下となるようにしたことを特徴とする単結晶の成長装置。

【請求項2】 上記種結晶支持部の上記側面を凸面状または凹面状とした請求項1記載の単結晶の成長装置。

【請求項3】 上記種結晶支持部の上記支持面の径を、上記種結晶の径と略同一または上記種結晶より小径とした請求項1または2のいずれか記載の単結晶の成長装置。

【請求項4】 上記単結晶が炭化珪素単結晶である請求項1ないし3のいずれか記載の単結晶の成長装置。

【請求項5】 容器内に、成長させる単結晶の原料と種結晶とを対向させて配置し、上記原料を加熱昇華させて上記種結晶上に単結晶を成長させる方法において、上記原料に対向する上記容器内壁面の一部を上記原料側に突出させて上記種結晶を支持する種結晶支持部となし、該種結晶支持部を、側面が傾斜する略台形形状とするとともに、この傾斜する側面の中央部における法線と上記支持面の法線とのなす角度 θ が20度以上80度以下となるようにしたことを特徴とする単結晶の製造方法。

【請求項6】 上記種結晶支持部の上記側面を凸面状または凹面状とした請求項5記載の単結晶の製造方法。

【請求項7】 上記種結晶支持部の上記支持面の径を、上記種結晶の径と略同一または上記種結晶より小径とした請求項5または6記載の単結晶の製造方法。

【請求項8】 上記単結晶が炭化珪素単結晶である請求項5ないし7のいずれか記載の単結晶の製造方法。

【発明の詳細な説明】**【0001】**

【発明の属する技術分野】 本発明は、炭化珪素等の単結晶を成長させるために用いられる単結晶の成長装置および製造方法に関する。

【0002】

【従来の技術】 炭化珪素単結晶は、熱的・化学的特性に優れ、パワーデバイス等の半導体装置作製用基板材料として注目されている。炭化珪素単結晶は、一般に、昇華再結晶法により得られ、成長させる単結晶の原料と種結晶とを対向させて配置し、原料を加熱昇華させて種結晶上に単結晶を成長させる。図5は、昇華再結晶法による成長装置の一例を示すもので、単結晶成長装置18は、炭化珪素原料粉末16が充填される黒鉛製のつぼ17と、該つぼ17に覆着される蓋体11を有している。蓋体11には、炭化珪素単結晶よりなる種結晶13が接

合固定してあり、原料粉末16を加熱昇華させると、その昇華ガスが対向する種結晶13上で再結晶して、炭化珪素単結晶15が成長する。

【0003】ところで、半導体装置作製の炭化珪素単結晶基板としては、現在、直径2インチ程度のものが市販されているが、量産性を向上させるために、より大口径の炭化珪素単結晶基板が必要とされている。ところが、上記図5に示した従来の成長装置18では、種結晶13が蓋体11に直接接合されるために、種結晶13周辺の蓋体11表面に堆積する多結晶14と、種結晶13上に成長する単結晶15とが接触する。このため、成長結晶の口径拡大が阻害される問題があった。

【0004】そこで、この問題を解決するための装置が種々提案されており、例えば、特公平6-37353号公報には、種結晶の周辺部を仕切り板で覆った成長装置が開示されている。この成長装置では、仕切り板を種結晶より高温に保持することにより、多結晶の発生を抑制し、種結晶上にのみ単結晶が成長するようにしている。また、特開平1-305898号公報、特開平10-36195号公報等に開示されるように、蓋体に突起部を形成して種結晶支持部とした成長装置がある。この装置では、図6のように、蓋体11の下面中央に形成した突起部を種結晶支持部12として、その上に種結晶13を配置しており、種結晶支持部12の周囲に堆積する多結晶14が種結晶13上に成長する単結晶15に接触するタイミングを遅らせて、成長結晶の口径拡大率を高めている。

【0005】

【発明が解決しようとする課題】 しかしながら、特公平6-37353号公報の装置では、得られる単結晶の口径が、仕切り板の開口径によって制限され、口径拡大率を十分大きくすることができない。また、装置構造が複雑で、装置の製作にコストと時間を要する問題がある。一方、特開平1-305898号公報、特開平10-36195号公報の装置では、多結晶が単結晶に接触するタイミングを遅らせることは可能であるものの、図6に示すように、成長が進むと、多結晶14が単結晶15に接触してしまうため、それ以上の口径の拡大が阻まれるという不具合があった。

【0006】 本発明は上記実情に鑑みてなされたものであり、その目的は、昇華再結晶法により炭化珪素等の単結晶を成長させるにあたり、多結晶による成長阻害を抑制して、簡便に、成長する単結晶の口径拡大を図ることができる単結晶の成長装置および製造方法を提供することにある。

【0007】

【課題を解決するための手段】 本発明の請求項1の単結晶の成長装置は、容器内に、成長させる単結晶の原料と種結晶とを対向させて配置し、上記原料を加熱昇華させて上記種結晶上に単結晶を成長させる装置であり、上記

原料に対向する上記容器内壁面の一部を上記原料側に突出させて、上記種結晶を支持する種結晶支持部となしてある。該種結晶支持部は、側面が傾斜する略台形形状であり、この傾斜する側面の中央部における法線と上記支持面の法線とのなす角度 θ を20度以上80度以下となるように形成してある。

【0008】上記装置を用いて単結晶を成長させると、上記種結晶支持部の側面上に多結晶が成長するが、本発明では上記側面を支持面側へ向けて縮径する傾斜面としたので、この傾斜する側面に成長する多結晶の成長方向はその法線の方向となる。すなわち、上記種結晶上に成長する単結晶の成長方向（上記支持面の法線の方向）に対して外側へ逃げるように多結晶が成長することになり、このため、蓋体に直に接合した従来構成（図5参照）のような多結晶による成長阻害を抑制することができる。特に、角度 θ を20度以上とすると、この効果が高く、単結晶が外側へ拡大しながら成長できるため、口径拡大率が大きくなる。

【0009】一方、凸状の種結晶支持部を設けた従来構成（図6参照）では、蓋体上に成長する多結晶が単結晶に接触するまで多結晶による成長阻害はないが、その間、単結晶の側面が高温に曝されるために、熱エッチングにより侵食されて口径拡大率が低下する。これに対し、本発明では、傾斜する上記側面上に単結晶成長の開始直後から多結晶が堆積し、これが成長する単結晶に接触して側面を保護する。この効果を得るには、角度 θ を80度以下とするのがよく、熱エッチングを防止して、口径を拡大させることができる。

【0010】従って、上記角度 θ を20度以上80度以下とすると、上記口径拡大効果が顕著であり、多結晶が単結晶の成長を妨害することなく、かつ成長する単結晶側面に接して熱エッチングを防止する。その結果、単結晶の口径拡大率を大幅に向上させることができ、しかも、装置構成が簡単で製作も容易である。

【0011】請求項2の装置では、上記種結晶支持部の上記側面を凸面状または凹面状に形成する。一般には、上記種結晶支持部を上記側面の断面が直線状となるように形成するが、上記側面を、略中央部が径方向外方に膨出する凸面状、あるいは径方向内方に凹陷する凹面状といった形状とすることもできる。この場合は、上記角度 θ が部位によって変化するが、上記支持面側端部または上記容器内壁面側端部を除く、少なくとも側面中央部における角度 θ が上記範囲にあれば、同様の効果が得られる。

【0012】請求項3の装置では、上記種結晶支持部の上記支持面の径を、上記種結晶の径と略同一または上記種結晶より小径とする。上記種結晶より上記支持面の径が大きいと、上記種結晶の周囲の上記支持面上に多結晶が堆積するので、傾斜する上記側面の効果を得るには、上記種結晶と上記支持面をほぼ同じ大きさとするか上記

支持面を若干小さく形成することが望ましい。

【0013】請求項4の装置では、上記単結晶を炭化珪素単結晶とする。炭化珪素単結晶は、半導体装置作製用基板として有用であり、しかも、従来、大口径化が困難であったので、本発明を適用した場合の効果が著しい。

【0014】請求項5の発明は、単結晶の製造方法に関するもので、容器内に、成長させる単結晶の原料と種結晶とを対向させて配置し、上記原料を加熱昇華させて上記種結晶上に単結晶を成長させる方法において、上記原料に対向する上記容器内壁面の一部を上記原料側に突出させて種結晶支持部となし、その上に上記種結晶を支持固定する。この時、該種結晶支持部を、側面が傾斜する略台形形状とし、この傾斜する側面の中央部における法線と上記支持面の法線とのなす角度 θ が20度以上80度以下となるようにするものである。

【0015】上記方法によれば、上記請求項1と同様の効果が得られ、簡易な方法で、単結晶の口径拡大率を大幅に向上させることができる。

【0016】請求項6の方法のように、上記種結晶支持部の上記側面を、凸面状または凹面状に形成することもできる。このようにしても、中央部における上記角度 θ が上記範囲となるようにすることで、いずれも同様の口径拡大効果が得られる。

【0017】請求項7の方法では、上記種結晶支持部の上記支持面の径を、上記種結晶の径と略同一または上記種結晶より小径とする。これにより、上記支持面上に多結晶が堆積するのを防止し、傾斜する上記側面の作用を効果的に発揮できる。

【0018】請求項8の方法では、上記単結晶を炭化珪素単結晶とする。炭化珪素単結晶は半導体装置作製用基板として有用であり、大口径化による利用価値が大きい。

【0019】

【発明の実施の形態】以下、図面に基づいて本発明の一実施の形態を詳細に説明する。図1(a)は、本発明を適用した単結晶の成長装置の概略構成図であり、ここでは、成長させる単結晶を炭化珪素単結晶とした場合について説明する。図中、単結晶の成長装置8は、容器としての黒鉛製のつぼ7および蓋体1を有している。つぼ7は、上端開口の容器体で、その下半部内には原料となる炭化珪素原料粉末6が充填してある。図1(b)に示すように、蓋体1は、外周縁部をやや薄肉のフランジ部1aとなしており、つぼ7に嵌着した時にフランジ部1aがつぼ7の上端縁に当接してこれを密閉するようになしてある（図1(a)）。

【0020】次に、本発明の特徴部分である蓋体1の形状について説明する。図1(a)、(b)において、蓋体1は、その下面（容器内壁面）中央を下方（原料粉末6側）に突出させて、種結晶3を支持固定するための種結晶支持部2となしている。この結晶支持部2は略台形

形状であり、側面2bが原料粉末6と傾きを有して対向するように、蓋体1下面側から種結晶3に接する支持面2a側へ向けて次第に縮径する形状となしてある。種結晶3が固定される支持面2aは、蓋体1下面と平行に形成される。

【0021】具体的には、側面2bを、支持面2aの法線Aと側面2bの法線Bとのなす角度 θ （以下、傾斜角度 θ という）が（図1（b））、20度以上80度以下となるよう傾斜させるのがよく、これにより、種結晶3の周囲に成長する炭化珪素多結晶4によって炭化珪素単結晶5の成長が阻害されるのを抑制し、かつ多結晶4により単結晶5の側面を熱エッチングから保護して、効果的に成長結晶5の口径拡大率を大きくすることができる。傾斜角度 θ が20度より小さいと、上記図6に示した従来構成に近くなり、周辺が多結晶による成長阻害が大きくなる。また、傾斜角度 θ が80度より大きいと、上記図7に示した従来構成に近くなり、単結晶外周部が熱エッチングされて、口径拡大効果が低減する。口径拡大効果が最大となる傾斜角度 θ は、周辺に成長する多結晶4の成長速度と単結晶5側面方向の成長速度の両者の大きさで決まる。両者の大きさは、その時の成長条件、成長装置の構造により変化するが、本発明者らは、図1（a）に示す構造の成長装置のおよび以下に示す成長条件の範囲であれば、傾斜角度 θ が20度から80度の範囲で口径拡大効果が得られることを見出した。より好ましくは、傾斜角度 θ を25度から65度の範囲とするのがよく、上記効果をさらに高めることができる。なお、本実施の形態では、結晶支持部2の側面2bを、断面が直線状となるように形成しており、傾斜角度 θ は一定である。

【0022】ここで、炭化珪素単結晶6を成長させるための種結晶3としては、通常、アチソン法で製造された炭化珪素単結晶、または、アチソン結晶から昇華法で成長させた炭化珪素単結晶が使用される。種結晶3は、これら炭化珪素単結晶を所定のウェハ形状に加工してなり、種結晶支持部2の支持面2a上に接合固定される。この時、支持面2aの径が種結晶3の径と略同一かあるいは若干小さくなるようにするのがよく、上記口径拡大効果をより効果的に発揮させることができる。支持面2aの径が種結晶3の径より大きすぎると、種結晶3周辺の支持面2a上に多結晶が堆積して、口径拡大に悪影響を及ぼすおそれがある。

【0023】なお、種結晶支持部2の突出高さ（図の上下方向の高さ）は、特に制限されないが、あまり小さすぎると、蓋体1上に成長する多結晶4が単結晶5の成長に追い付いてしまい、種結晶支持部2がない状態（傾斜角度 $\theta=0$ の状態）に近くなる。また、突出高さが大きすぎると、種結晶3まわりの温度分布が変化するなどの問題があるため、装置形状や種結晶3の大きさ等に応じて、適宜設定するのがよい。

【0024】上記装置を用いて単結晶を成長させる場合には、図1（a）のように、種結晶3を、結晶支持部2の支持面2aに接着剤によって接合し、るつぼ7の周囲に配した誘導コイル等の加熱装置（図略）で加熱する。この時、原料粉末6が炭化珪素の昇華温度以上（通常、約2000～2500℃程度）、種結晶3が原料粉末6より低い温度となるように、るつぼ7内に温度勾配を設ける。るつぼ7内の雰囲気は、アルゴンガス等の不活性ガス雰囲気とし、圧力は0.1～10 Torr程度とするのがよい。これにより、原料粉末6の昇華ガスが発生して上方へ拡散し、より低温の種結晶3上で再結晶する。

【0025】このように、結晶支持部2の側面2bを傾斜面とし、その傾斜角度 θ （支持面2aの法線Aと側面2bの法線Bとのなす角度）を20度以上80度以下とすることで、得られる炭化珪素単結晶6の口径拡大角度 α （単結晶5と多結晶4の境界線Cと支持面2aの法線Aとのなす角度）を従来より大きくすることが可能である。これは、傾斜する側面2bの法線Bの方向に成長する多結晶4が、種結晶3上に成長する単結晶5の成長方向（上記支持面の法線の方向）に対して外側へ逃げるように成長することになるために、多結晶4による成長阻害が防止されること、しかも、この多結晶4によって単結晶5の側面が保護されるために、熱エッチングが防止されることにより、これら両方の効果で、簡易に、成長する単結晶5の口径拡大を図ることができる。

【0026】図2に本発明の第2の実施の形態を示す。上記第1の実施の形態において、結晶支持部2の側面2bは断面が直線状となるように形成され、傾斜角度 θ は一定であるが、図2のように、側面2bを、略中央部が径方向外方に膨出する凸面状とすることもできる。この場合は、支持面2a側から蓋体1側へ向けて、傾斜角度 θ が徐々に大きくなるが、支持面2a側端部および蓋体1側端部を除く中央部において、傾斜角度 θ が20度以上80度以下となるようにすることで、同様の効果が得られる。また、図3に本発明の第3の実施の形態を示すように、側面2bを、略中央部が径方向内方に凹陷する凹面状に形成することもできる。この場合も同様で、支持面2a側から蓋体1側へ向けて、傾斜角度 θ が徐々に小さくなるが、支持面2a側端部および蓋体1側端部を除く中央部において、傾斜角度 θ が20度以上80度以下となるようにすることで、同様の効果が得られる。

【0027】また、結晶支持部2および種結晶3の形状は、円形が一般的であるが、円形に限らず、他の形状、例えば、四角形、六角形等の多角形とすることもできる。この時、結晶支持部2と種結晶3の形状を一致させる必要は必ずしもなく、例えば、多角形の種結晶3を円形の結晶支持部2上に支持させたり、逆に、円形の種結晶3を多角形の結晶支持部2上に支持させることもできる。

【0028】

【実施例】本発明の効果を確認するために、上記図1の単結晶成長装置8を用いて炭化珪素単結晶5を成長させた。種結晶3としてはアチソン結晶を用い、これを直径10mmのウエハ状に成形した後、蓋体1に設けた種結晶支持部2の支持面2aに、接着剤を用いて貼り付けた。種結晶支持部2は、支持面2aの外径が10mm、傾斜角度 θ （支持面2aの法線Aと側面2bの法線Bとのなす角度）が30度となるように形成した。るつぼ7内に、炭化珪素原料粉末6として市販の炭化珪素研磨材（平均粒子径80 μ m）を充填し、その上端開口に種結晶3を貼り付けた蓋体1を嵌着固定して、図略の成長用炉中で加熱した。この際、まず、るつぼ7内を図略の真空排気系にてアルゴンガス雰囲気置換し、雰囲気圧力700Torrで、るつぼ7内温度を成長温度まで上昇させた後、雰囲気圧力を成長圧力（10Torr）まで減圧して、成長を開始した。加熱は図略の誘導コイルによる誘導加熱方式とし、原料粉末6を約2200～2300℃に加熱して、これより低い温度に保持した種結晶3上に単結晶5を成長させた。成長時間は24時間とし、成長中は、温度と圧力を一定に保った。成長終了時は、まず、雰囲気圧力を700Torrまで上げてから、温度を常温まで低下させた。

【0029】その後、成長用炉からるつぼ7を取り出し、蓋体1を取り外して種結晶3上に成長した炭化珪素単結晶5の評価を行った。評価は、得られた炭化珪素単結晶5を縦方向（図1の上下方向）に切断して撮影した縦切断面の写真を用いて行い、炭化珪素単結晶5と炭化珪素多結晶4の境界線Cと、支持面2aの法線Aのなす角度を測定して、口径拡大角度 α とした。さらに、種結晶支持部2の傾斜角度 θ を15度から90度の範囲で変更し、同様の方法で成長させた炭化珪素単結晶5について、口径拡大角度 α をそれぞれ測定した。これら測定結果に基づき、傾斜角度 θ と口径拡大角度 α の関係を調べて、図4に示した。なお、この時、同じ傾斜角度 θ で複数の単結晶5を成長させ、各単結晶5の縦切断面のサンプルを作成して、その左右の口径拡大角度 α をそれぞれ測定した。図4には、口径拡大角度 α を、このサンプルの部位によるばらつきおよびサンプル間のばらつきの幅とともに示した。

【0030】図4に明らかなように、傾斜角度 θ が15度から大きくなるに従い、口径拡大角度 α が増大しており、傾斜角度 $\theta=20$ 度で口径拡大角度 α が30度以上、傾斜角度 $\theta=25$ 度で口径拡大角度 α が40度以上の、口径拡大効果が得られる。すなわち、傾斜角度 θ を大きくすることで、種結晶支持部2の側面2b上に成長

する多結晶4の成長方向がより外側に傾くことから、単結晶5の成長が阻害されず、単結晶5がより外側に拡大しながら成長できることがわかる。これに対し、図5の従来装置構成に近い傾斜角度 $\theta=15$ 度では、十分な効果が得られず、単結晶5に近接して成長する多結晶4による成長阻害が生じていると考えられる。

【0031】一方、傾斜角度 θ が40度を越えると、口径拡大角度 α が徐々に小さくなり、図6の従来装置構成となる傾斜角度 $\theta=90$ 度では、口径拡大角度 α が25度程度となる。これは、傾斜角度 θ が大きくなりすぎると、単結晶5と多結晶4が接しないために、単結晶5の側面が露出して熱エッチングを受けるためと考えられ、側面2b上の多結晶4が単結晶5の成長を妨げない程度にこれに接することで、単結晶5の熱エッチングを防止する効果が得られることがわかる。以上より、側面2b上の多結晶4が単結晶5の成長を妨げず、かつ単結晶5の側面を熱エッチングから保護するには、傾斜角度 θ が20度から80度の範囲にあればよく、従来に比べて大幅に口径を拡大できることがわかる。

【0032】なお、上記実施の形態では、単結晶として炭化珪素単結晶を成長させた場合について説明したが、炭化珪素単結晶以外にも、昇華再結晶法で成長可能な他の単結晶の成長に適用してももちろんよい。

【図面の簡単な説明】

【図1】本発明の第1の実施の形態を示し、(a)は単結晶成長装置の概略断面図、(a)は蓋体の拡大断面図である。

【図2】本発明の第2の実施の形態における蓋体の拡大断面図である。

【図3】本発明の第3の実施の形態における蓋体の拡大断面図である。

【図4】角度 θ と口径拡大角度 α の関係を示す図である。

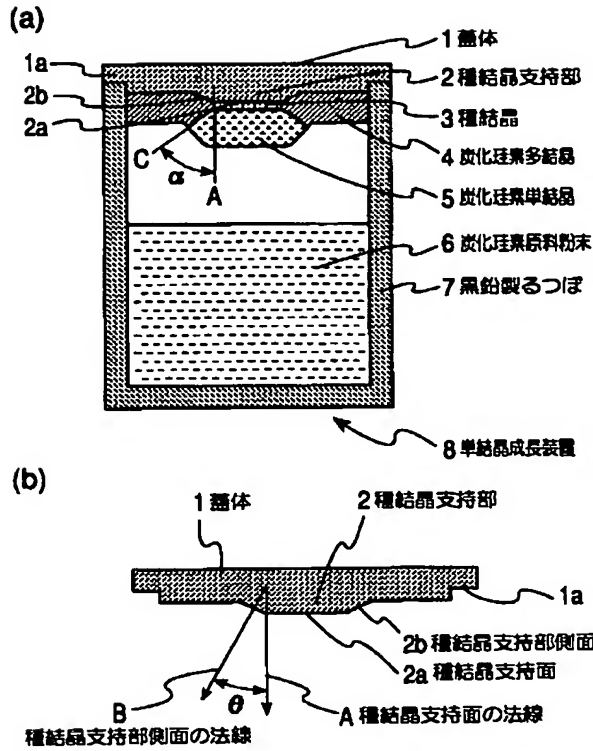
【図5】従来の単結晶成長装置の概略断面図である。

【図6】従来の単結晶成長装置の概略断面図である。

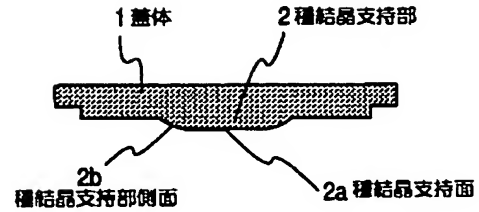
【符号の説明】

- 1 蓋体
- 2 種結晶支持部
- 2a 支持面
- 2b 側面
- 3 種結晶
- 4 炭化珪素多結晶
- 5 炭化珪素単結晶
- 6 原料粉末
- 7 るつぼ
- 8 単結晶成長装置

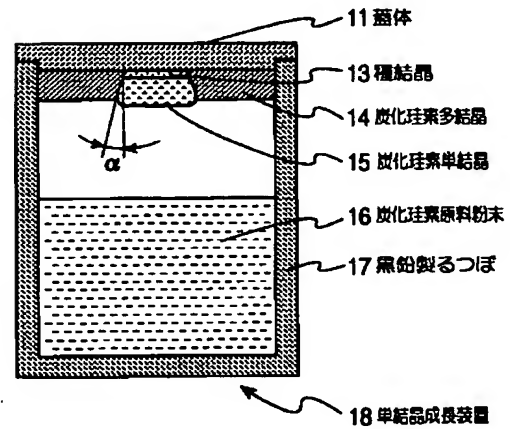
【図 1】



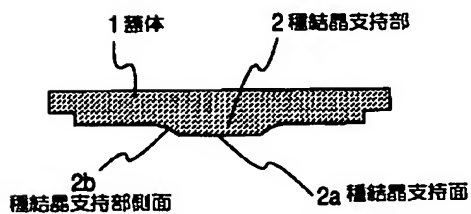
【図 2】



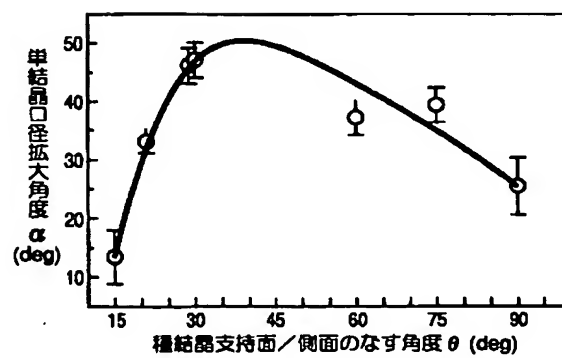
【図 5】



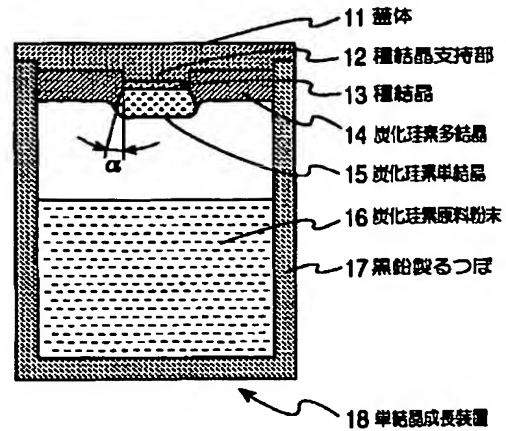
【図 3】



【図 4】



【図6】



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Fターム(参考) 4G077 AA02 BE08 DA02 DA18 EG11